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# GAS ADSORPTION BY ACTIVATED AND IMPREGNATED CARBONS

## QUARTERLY PROGRESS REPORT

October 1976 to January 1977

by

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

In the period, equilibrium adsorption isotherms have been measured for $H_2O$ vapor on BPL activated and ASC whetlerite impregnated carbons. Work was also initiated on adsorption of binary mixed vapors by the carbons. It was found that HCN adsorption by BPL carbon in equilibrium with water vapor at a relative pressure of 0.8 produced a total adsorption of $H_2O$ and HCN which was greater than the adsorption of any previously measured single adsorbate. However, when the same carbon, a new BPL carbon sample or an ASC carbon sample was exposed to the same mixture, much smaller adsorption was noted.

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## PREFACE

The work described in this report was authorized under Contract No. DAAA 15-74-C-0163. Task 1W762710A09501, Gas Adsorption by Activated and Impregnated Carbons. The work described covers the period from 9 October 1976 to 8 January 1977.

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CONTENTS

## RESEARCH PROGRESS DURING THE PERIOD

### I. INTRODUCTION

In this period, $H_2O$ vapor equilibrium adsorption isotherms were determined for BPL activated and ASC whetlerite carbons with water vapor relative pressures ranging from 0.005 up to 0.95. Emphasis was placed on the high pressure region of the isotherm. Studies were initiated on the adsorption of binary mixed vapors by activated and impregnated carbons. BPL activated carbon in equilibrium with relative water vapor pressures ~0.8 was exposed to HCN and allowed to equilibrate. BPL activated and ASC whetlerite carbons were also exposed to $HCN-H_2O$ mixtures.

### II. ADSORPTION OF WATER VAPOR BY BPL ACTIVATED AND ASC WHETLERITE CARBONS

The outgassing procedure for these carbons consisted of heating the carbon to 400°C (150°C for ASC whetlerite) under $10^{-6}$ torr and vacuum pumping until the sample reached a constant minimum weight. The sample was then cooled to room temperature under vacuum, exposed to purified water vapor, and allowed to equilibrate. This procedure was repeated as subsequent isotherm points were obtained. At the end of an experimental run, the sample was again heated to 400°C (150°C for ASC whetlerite) under $10^{-6}$ torr and vacuum pumped until a constant minimum weight was reached. The sample mass was then recorded and compared to the initial minimum sample weight. Any adsorbate remaining after this process was attributed to chemically bound or chemisorbed material.

### III. WATER VAPOR ADSORPTION RESULTS AND DISCUSSION

Isotherm data were plotted in the form of the Dubinin-Polanyi equation (figures 1 and 2):

$$\log W = \log W_0 - k\,\varepsilon^2$$

where W, $W_0$, k and $\varepsilon$ have been defined previously[1]. The isotherm data were also plotted in the conventional form (W vs $P/P_0$) where P is the equilibrium pressure of adsorbate vapor and $P_0$ is the saturated vapor pressure of adsorbate liquid vapor (Figures 3 and 4).

As was noted earlier[2] a great deal of hysteresis is exhibited by the adsorption-desorption process of water vapor by both ASC whetlerite and BPL activated carbons. This hysteresis does not seem to be reconciled with any pressure range other than the relative pressure end points of zero and one. This situation, coupled with the long equilibration times at higher pressures (several hours), makes it very difficult to define one smooth definitive isotherm, and the actual amount of adsorbate at a particular relative vapor pressure and temperature is, of course, dependent on sample history which in turn is perturbed by changes in the ambient temperature.

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[1] P. J. Reucroft, W. H. Simpson, and L. A. Jonas, J. Phys. Chem., 75, 3526 (1971).

[2] P. J. Reucroft, C. P. Madhusudhan and G. B. Freeman, Tenth Quarterly Progress Report, July 1976 to October 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

It was reported earlier[3] that both carbons adsorbed far less water than previously studied organic adsorbates at the same relative pressures. This is found to be true in the lower pressure portions of isotherms, while the final amounts of water vapor adsorbed can be seen (see figures 1 and 2) to compare very favorably with the amounts of saturation adsorption shown by the previously studied organic adsorbates[4].

## IV. ADSORPTION OF HCN AND $H_2O$ VAPOR MIXTURES BY BPL ACTIVATED AND ASC WHETLERITE CARBONS

The procedure followed was as follows:

1. A BPL sample was outgassed as described in section II of this report and the minimum sample mass was recorded.

2. The BPL sample was allowed to equilibrate with $H_2O$ vapor at a relative vapor pressure near to 0.8.

3. Purified HCN was then introduced into the system already in equilibrium with the water vapor. The water and HCN mixture was initially assumed to be non-reactive and the initial partial pressures of each gas was used to determine the HCN to $H_2O$ ratio in the system. Any additional adsorption was assumed to be due to adsorption of HCN.

4. The $H_2O$ and HCN was collected in a cold trap and the BPL activated carbon was outgassed at 400°C under the pumping power of the cold trap at liquid nitrogen temperature. The collected vapor mixture was then isolated from the BPL sample after a constant minimum sample weight was reached. Any adsorbate left when this weight was compared to the original minimum sample weight was ascribed to chemisorption.

5. The same sample was re-exposed to the same vapor mixture and step 4 was repeated.

6. The old sample was removed and a fresh BPL sample was outgassed as described in step 1 and step 5 was repeated for this sample.

7. The old sample was removed and a fresh ASC whetlerite sample was outgassed as in step 1 (but at 150°) and step 5 was repeated for this sample.

## V. HCN AND $H_2O$ MIXTURE ADSORPTION RESULTS AND DISCUSSION

The data from this portion of the report is displayed in tables 3 and 4 in chronological order within a particular data run. The initial water vapor relative pressures were between 0.82 and 0.91 which, from the water vapor adsorption data, indicates that the water adsorbed is very close to saturation. That is, the greatest observed amount of water vapor adsorbed on a BPL activated carbon has been 0.407 $cm.^3g^{-1}$, a value which has not been exceeded even under conditions of 100% humidity. When the BPL samples in equilibrium with water vapor were exposed to HCN vapor, a significant adsorption on the order of 0.07 $cm.^3g^{-1}$ was noted bringing the total amount of adsorbate to 0.42 $cm.^3g^{-1}$ in data run #1, to 0.47 $cm.^3g^{-1}$ in run #2 and to 0.48

[3] Ibid.

[4] P. J. Reucroft and C. T. Chiou, Fourth Quarterly Progress Report, January 1975 to April 1975, Edgewood Arsenal Contract DAAA15-74-C-0163.

$cm.^3g^{-1}$ in data run #3. This is less than the amount of adsorbate expected if it is assumed that the total amount of adsorbate would be the sum of the individual processes. For example, from Table 3, in run #2, at a relative water vapor pressure of 0.91 $W_{H_2O}$ was equal to 0.39 $cm.^3g^{-1}$ and at a relative HCN vapor pressure of 0.08 $W_{HCN}$ should be equal to 0.17 $cm.^3g^{-1}$ [5] giving a total adsorption equal ot 0.56 $cm.^3g^{-1}$. In actuality, the total adsorption was about 0.47 $cm.^3g^{-1}$; less than expected from the vapors acting completely independently and substantially greater than the $W_0$ values measured for BPL in past reports.

Chemisorption data can be found in Table 4 where the chemically bound remains ranged from 0.0069 to 0.0000 $cm.^3g^{-1}$ for the HCN-$H_2O$ mixture on BPL. It is believed that these amounts do not actually reflect true chemisorption, but rather the limited pumping power of the cold trap compared to the sorption and ion pumps used initially to define the dry mass of the sample. The process here may be accounted for by physical adsorption.

When the HCN and $H_2O$ were withdrawn from the sample, the resultant pressure in the portion of the system isolated from the sample was less than that expected from the partial pressures of the individual gases. This fact, coupled with the relatively small amount of adsorption measured when the same BPL sample or a new BPL sample was exposed to that same HCN-$H_2O$ mixture, seems to point to a strong possibility of chemical activity between the HCN and $H_2O$.

To summarize, after the BPL samples were allowed to equilibrate with water and then exposed to HCN, the total amount of adsorbate was unusually large. However, upon re-exposure to the same amount of $H_2O$-HCN, now in a mixture, the BPL sample adsorbed a significantly smaller mass of adsorbate, and most probably a significantly smaller volume of adsorbate. This, however, does not imply a loss of adsorptivity by the BPL sample, because in run #1 a new BPL sample showed essentially the same reaction to the $H_2O$-HCN mixtures as that of the re-exposed BPL samples.

The new ASC whetlerite impregnated carbon samples examined actually adsorbed less of the $H_2O$-HCN mixture than did the BPL samples. It is not clear at this time whether this comparison is significant. However, it is clear that the amount adsorbed by the ASC whetlerite was similar to that of the BPL samples exposed to the mixture and significantly less than the total amounts adsorbed by the BPL samples already in equilibrium with water vapor.

The ASC whetlerite samples did exhibit clearly significant amounts of chemisorption and thus prohibited any further work with that $H_2O$-HCN mixture because the desorbed mixture clearly was no longer the same as it had been and there was no way to determine the new physical or chemical makeup of that $H_2O$-HCN mixture.

The high relative water vapor pressures proved to be hazardous to the microbalance phototube when a relative pressure of 1 was approached too closely. Both balances were temporarily disabled confirming this effect. This effect has been reduced with the addition of a small heat lamp to irradiate the balance. This appears to reduce condensation of water vapor on a glass enclosed high impedance resistor. Another difficulty concerns the design of the system. The constituent ratio of the gaseous mixture cannot be easily varied in the present system. This problem will be alleviated in the near future with the addition of another storage

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[5] P. J. Reucroft and C. T. Chiou, Seventh Quarterly Progress Report, October 1975 to January 1976, Edgewood Arsenal Contract DAAA15-74-C-0163.

flask and it should be possible to measure portions of the isotherms during one experimental run and not be limited to determination of a single isotherm point. The data thus far has dealt with samples in equilibrium with $H_2O$ exposed to HCN and samples exposed to the $H_2O$-HCN mixtures which had previously been adsorbed on BPL carbons. In the next period, situations will be examined where carbons are exposed to fresh $H_2O$-HCN mixtures (in varying amounts) and where samples in equilibrium with HCN are then exposed to $H_2O$ vapor.

The most serious problem is the inability to detinitively state a) which gas is being adsorbed from the gaseous mixtures, b) whether or not any desorption of one gas is taking place while the other gas is being adsorbed, and c) whether the gaseous mixture is indeed retaining its integrity or whether chemical reactions are taking place in the gaseous mixture. It is not possible to analyze the gaseous mixture in the system at this time and definitive statements concerning mass changes in the sample can only be made.

Appendix I

Tables

TABLE 1. ADSORPTION AND DESORPTION DATA

Adsorbate: Water Vapor $\rho$ = 0.998

Adsorbent: BPL Activated Carbon

| | Sample I Adsorption | | | Sample II Adsorption | |
|---|---|---|---|---|---|
| $P/P_0$ | $\epsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) | $P/P_0$ | $\epsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) |
| 0.024 | 4.774 | 0.002 | 0.495 | 0.170 | 0.071 |
| 0.071 | 2.402 | 0.003 | 0.558 | 0.119 | 0.128 |
| 0.147 | 1.265 | 0.005 | 0.597 | 0.093 | 0.191 |
| 0.260 | 0.624 | 0.009 | 0.612 | 0.084 | 0.272 |
| 0.323 | 0.441 | 0.013 | 0.652 | 0.064 | 0.262 |
| 0.403 | 0.282 | 0.024 | 0.693 | 0.047 | 0.306 |
| 0.457 | 0.210 | 0.035 | 0.765 | 0.025 | 0.334 |
| 0.500 | 0.165 | 0.050 | 0.829 | 0.012 | 0.363 |
| 0.523 | 0.145 | 0.071 | 0.862 | 0.008 | 0.368 |
| 0.544 | 0.128 | 0.095 | | | |
| 0.554 | 0.120 | 0.149 | | | |
| 0.627 | 0.075 | 0.220 | | | |
| 0.650 | 0.064 | 0.255 | | | |
| 0.705 | 0.042 | 0.302 | | | |
| 0.775 | 0.022 | 0.342 | | | |
| 0.812 | 0.015 | 0.359 | | | |
| 0.863 | 0.008 | 0.372 | | | |
| 0.903 | 0.004 | 0.384 | | | |
| 0.942 | 0.001 | 0.394 | | | |

TABLE 1. Contd.

Adsorbate: Water Vapor $\rho$ = 0.998

Adsorbent: BPL Activated Carbon

Sample III
Adsorption

| $P/P_0$ | $\epsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) |
|---|---|---|
| 0.684 | 0.050 | 0.296 |
| 0.833 | 0.011 | 0.376 |

Sample III
Desorption

| $P/P_0$ | $\epsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) |
|---|---|---|
| 0.838 | 0.011 | 0.376 |
| 0.764 | 0.025 | 0.371 |
| 0.719 | 0.038 | 0.362 |
| 0.684 | 0.050 | 0.296 |
| 0.663 | 0.058 | 0.346 |
| 0.618 | 0.080 | 0.333 |
| 0.607 | 0.085 | 0.319 |
| 0.560 | 0.116 | 0.313 |
| 0.543 | 0.128 | 0.291 |
| 0.532 | 0.135 | 0.174 |
| 0.483 | 0.181 | 0.057 |
| 0.371 | 0.337 | 0.013 |

TABLE 2. ADSORPTION DATA

Adsorbate: Water Vapor $\rho = 0.998$

Adsorbent: ASC Whetlerite Carbon

| Sample I | | | Sample II | | |
|---|---|---|---|---|---|
| $P/P_0$ | $\varepsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) | $P/P_0$ | $\varepsilon^2 \times 10^{-6}$ ($cal.^2 mole^{-2}$) | W ($cm.^3 g^{-1}$) |
| 0.258 | 0.637 | 0.043 | 0.037 | 3.731 | 0.016 |
| 0.331 | 0.425 | 0.057 | 0.153 | 1.218 | 0.029 |
| 0.389 | 0.311 | 0.081 | 0.264 | 0.613 | 0.041 |
| 0.442 | 0.232 | 0.125 | 0.336 | 0.410 | 0.058 |
| 0.468 | 0.199 | 0.158 | 0.395 | 0.298 | 0.086 |
| 0.533 | 0.138 | 0.186 | 0.424 | 0.253 | 0.121 |
| 0.568 | 0.112 | 0.211 | 0.485 | 0.181 | 0.150 |
| 0.623 | 0.078 | 0.251 | 0.529 | 0.140 | 0.188 |
| 0.634 | 0.073 | 0.293 | 0.584 | 0.100 | 0.230 |
| 0.770 | 0.024 | 0.315 | 0.631 | 0.073 | 0.287 |
| 0.837 | 0.011 | 0.335 | 0.740 | 0.031 | 0.312 |
| 0.905 | 0.004 | 0.358 | 0.815 | 0.015 | 0.335 |
| | | | 0.865 | 0.007 | 0.350 |

## TABLE 3. GASEOUS MIXTURE ADSORPTION DATA

| New BPL Sample | Run #1 | Run #2 | Run #3 |
|---|---|---|---|
| Dry Mass[a] | 224.98 | 240.76 | 239.64 |
| **Water Vapor Admitted** | | | |
| Water Pressure[b] | 0.82 | 0.91 | 0.825 |
| $T^c$ | 22.6 | 23.0 | 22.9 |
| Mass | 304.98 | 333.84 | 326.72 |
| $\epsilon^2 \times 10^{-6}$ | 0.014 | 0.0032 | 0.013 |
| $W^d_{H_2O}$ | 0.356 | 0.388 | 0.407 |
| **HCN Admitted in the Presence of Water** | | | |
| HCN Pressure | 0.082 | 0.080 | 0.053 |
| T | 23.2 | 24.2 | 23.2 |
| Mass | 315.2 | 344.64 | 338.4 |
| $\epsilon^3 \times 10^{-6}$ | 2.15 | 2.23 | 3.02 |
| $W^e_{HCN}$ | 0.066 | 0.078 | 0.072 |
| $W^f_{HCN}$ | 0.19 | 0.17 | 0.15 |
| **Water and HCN Removed** | | | |
| Mass | 225.34 | 242.04 | 239.98 |
| **Water and HCN Readmitted** | | | |
| T | 23.8 | 24.0 | 23.5 |
| Mass | 254.1 | 279.20 | 285.10 |
| $\rho^g$ Mixture | 0.80 | 0.79 | 0.81 |
| $W^h$ | 0.162 | 0.202 | 0.240 |

TABLE 3 (Contd.)

| | | | |
|---|---|---|---|
| Water and HCN Removed | | | |
| Mass | 224.98 | 242.00 | 239.84 |
| Water and HCN Readmitted | | | |
| T | 23.1 | --- | --- |
| Mass | 257.1 | --- | --- |
| ρ | 0.80 | --- | --- |
| W | 0.178 | --- | --- |
| Water and HCN Removed | | | |
| Mass | 225.00 | --- | --- |
| New BPL Sample | | | |
| Dry Mass | --- | 245.83 | 241.01 |
| Water and HCN Readmitted | | | |
| T | --- | 23.2 | 22.2 |
| Mass | --- | 284.80 | 276.8 |
| ρ | --- | 0.79 | 0.81 |
| W | --- | 0.201 | 0.183 |
| Water and HCN Removed | | | |
| Mass | --- | 245.86 | 241.48 |
| New ASC Whetlerite Sample | | | |
| Dry Mass | 254.48 | --- | 225.76 |
| Water and HCN Admitted | | | |
| T | 24.3 | --- | 24.4 |
| Mass | 283.96 | --- | 257.2 |
| ρ | 0.80 | --- | 0.81 |
| W | 0.145 | --- | 0.171 |

TABLE 3 (Contd.)

| | | | |
|---|---|---|---|
| Water and HCN Removed | | | |
| Mass | 258.64 | --- | 229.68 |

a All masses are in mg.

b All pressures are relative pressures $P/P_0$ for that gas

c Temperatures are given in °C

d $W_{H_2O}$ is the $H_2O$ adsorbed in $cm.^3g^{-1}$

e $W_{HCN}$ is the HCN adsorbed in $cm.^3g^{-1}$ assuming no $H_2O$ is desorbed

f $W^f_{HCN}$ is the HCN which would be adsorbed with a clean sample of the same dry mass at the same value of $\epsilon^2$ (i.e. in the absence of water)

g It is assumed, as a starting point, that the HCN and $H_2O$ molecules do not chemically react and that the material adsorbed retains the same molecular ratio as present in the gaseous phase

h W is the HCN-$H_2O$ mixture adsorbed assuming the densities described in g

TABLE 4. CHEMISORPTION [g]

| Adsorbent | Data Run | Adsorbate | $W_{CH}$ ($cm.^3g^{-1}$)[f] |
|---|---|---|---|
| BPL[a] | 1 | HCN on $H_2O$ | 0.0020 |
| BPL[a] | 1 | $H_2O$ and HCN | 0.0000 |
| BPL[a] | 1 | $H_2O$ and HCN | 0.0001 |
| BPL[b] | 2 | HCN on $H_2O$ | 0.0069 |
| BPL[b] | 2 | $H_2O$ and HCN | 0.0067 |
| BPL[d] | 2 | $H_2O$ and HCN | 0.0002 |
| BPL[c] | 3 | HCN on $H_2O$ | 0.0017 |
| BPL[c] | 3 | $H_2O$ and HCN | 0.0010 |
| BPL[e] | 3 | $H_2O$ and HCN | 0.0024 |
| ASC | 1 | $H_2O$ and HCN | 0.0204 |
| ASC | 3 | $H_2O$ and HCN | 0.0214 |

a Each sample superscripted with the same letter is the same sample

f $W_{CH}$ is the volume of adsorbate chemically retained assuming an adsorbate density of 0.8 $g.cm.^{-3}$

g In each case, the chemisorption is computed with respect to the dry mass (minimum sample mass) determined for each sample before exposure to the adsorbents.

Appendix II

Figures

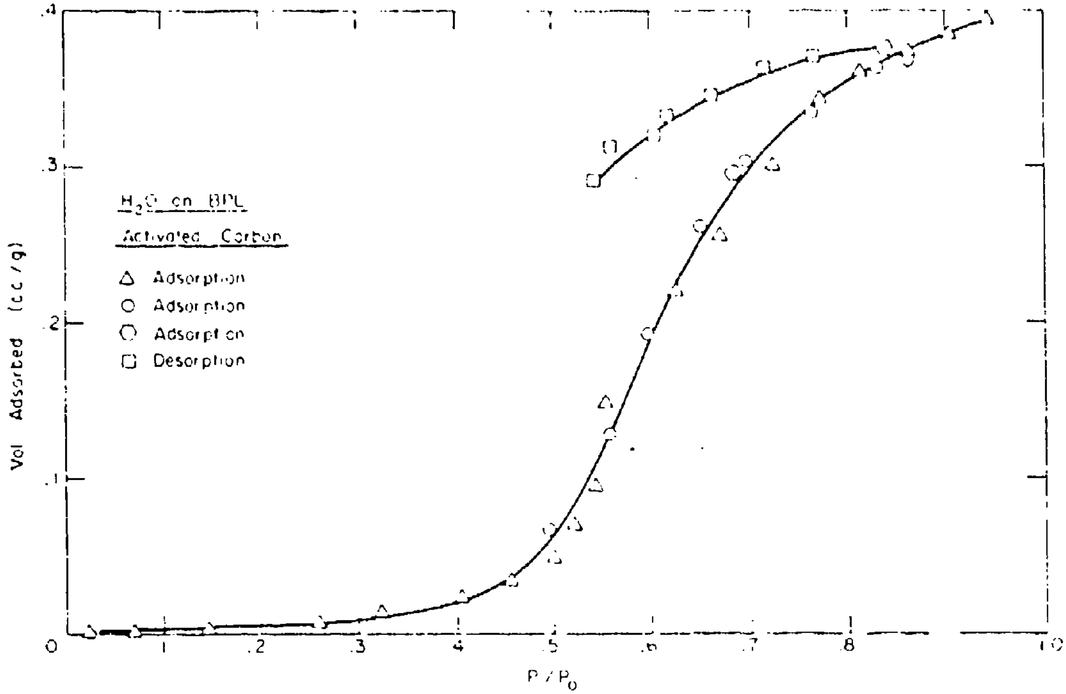


Figure 1. $H_2O$ on BPL Activated Carbon

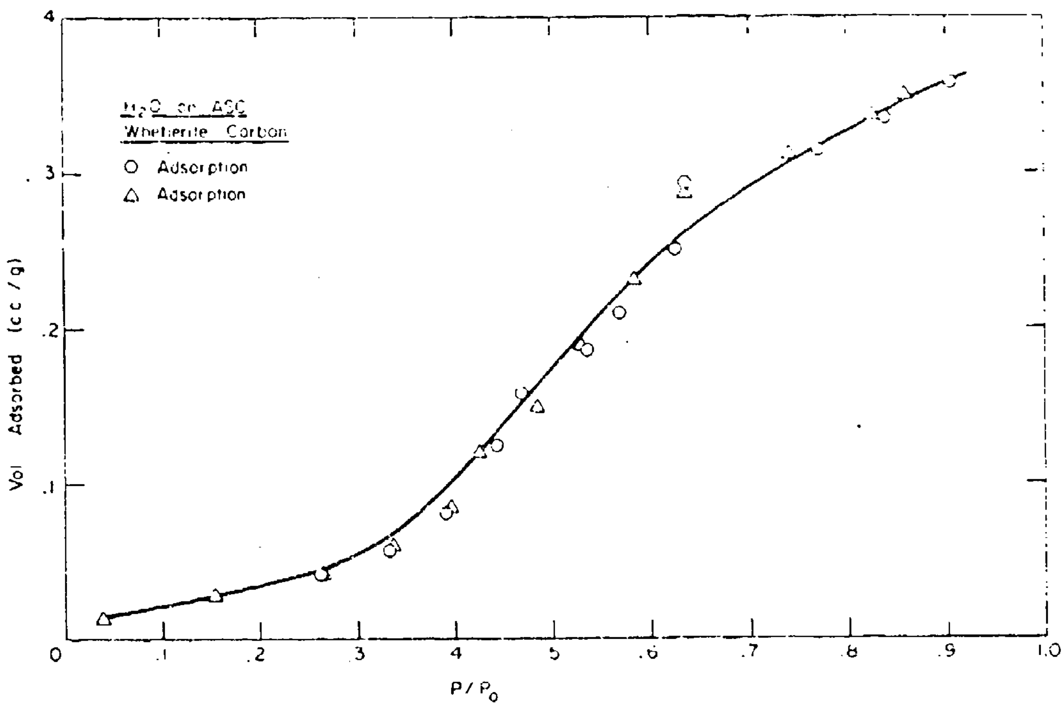


Figure 2. $H_2O$ on ASC Whetlerite Carbon

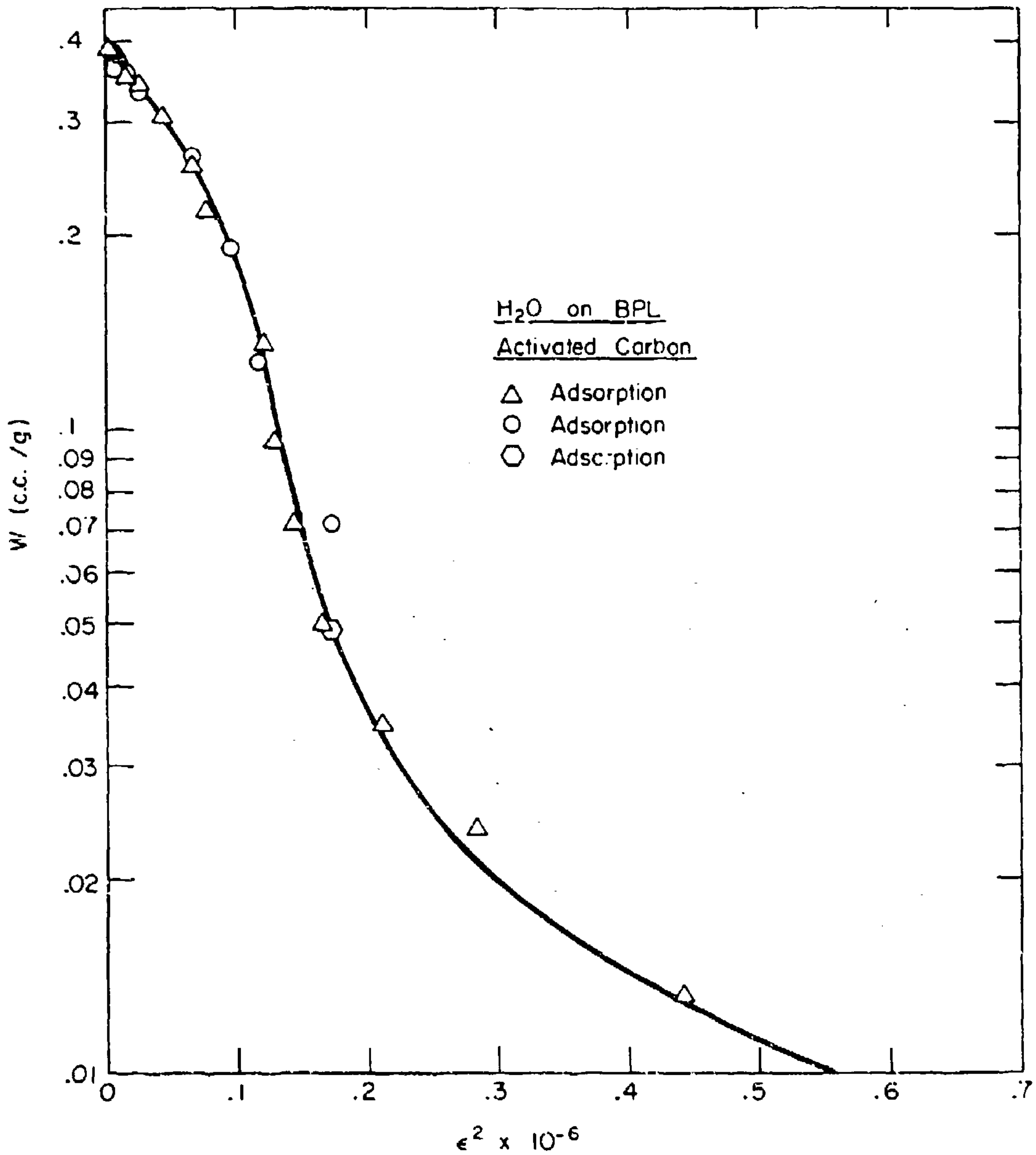


Figure 3. Adsorption and Desorption Isotherm of $H_2O$ on BPL Activated Carbon

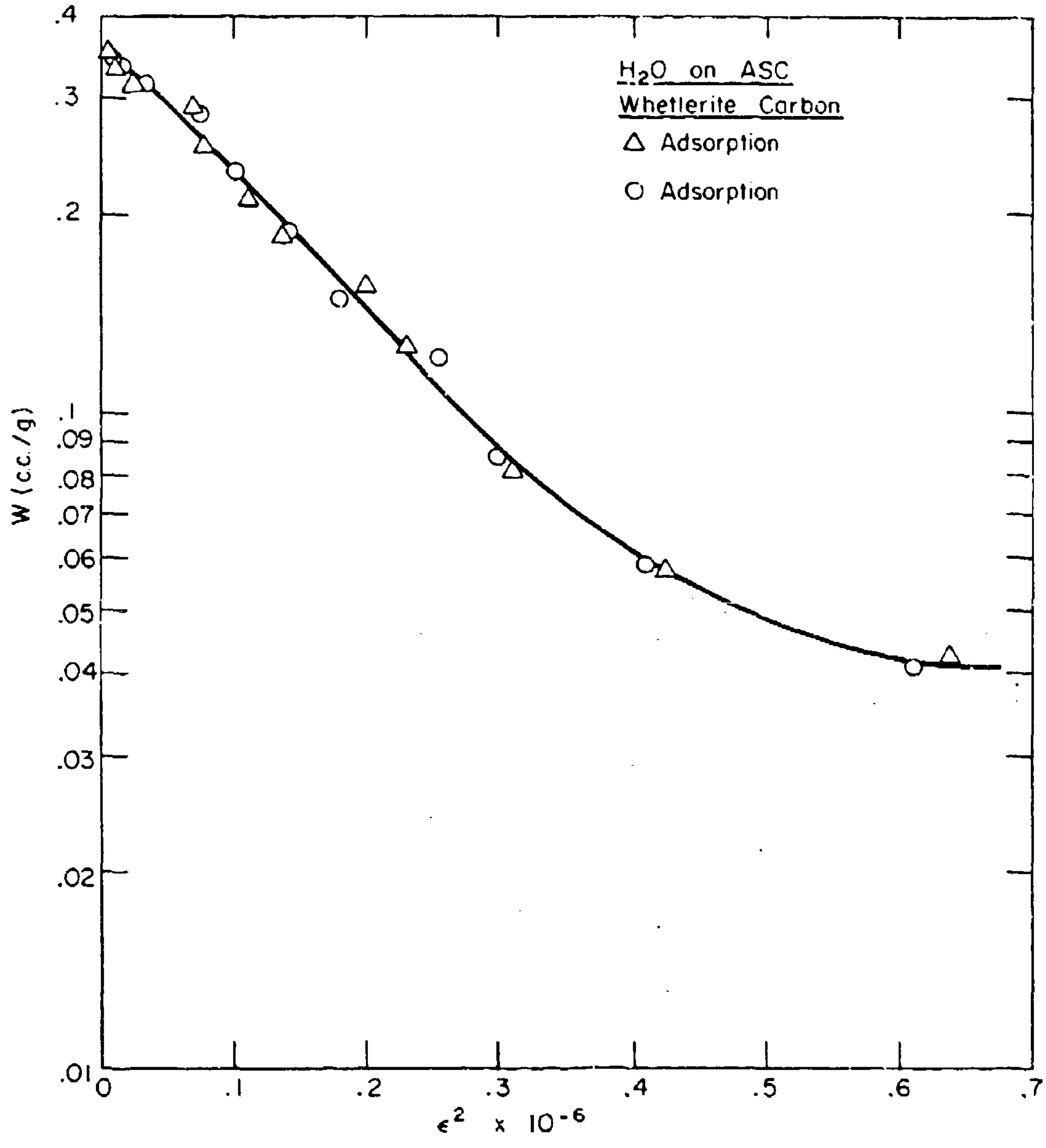


Figure 4. Adsorption Isotherm of $H_2O$ on ASC Whetlerite Carbon

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